A New Dynamical Diffraction-Based Technique of Residual Stress Measurements in Thin Films

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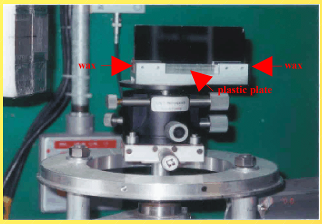
The residual stress in a thick Si(111) crystal coated with a 2000 Å thick Ni film was measured using a newly discovered dynamical diffraction effect "the Neutron Camel". The radius of bending is ~ 19 km and the corresponding tension force applied to the Ni film is 90 ± 5 N/m.

Introduction

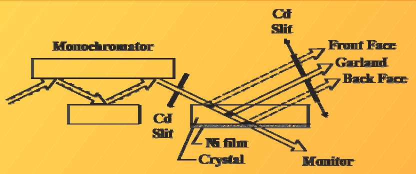
In the past two decades, optical devices, consisting of thin reflecting layers deposited on thick silicon or silicon dioxide substrates, have found wide application in light, X-ray and neutron diffraction. A significant surface-induced residual stress which usually remains in the films as well as in the substrates after deposition, creates a serious limitation of quality of these devises. The residual stress in crystalline films can be detected directly by the conventional X-ray diffraction technique. There exists another laser-based *in situ* technique, Surface-Stress-Induced Optical Deflection (SSIOD), which is able to detect small deformation strain in substrates during the coating process. The Back-Face Neutron Diffraction (BFND) from a perfect Si crystal, as it was shown in our previous study, is extremely sensitive to the ultra-small deformation strain. This technique is capable of detecting residual stress in single crystals even when the relative deformation of the crystallographic cells is as small as $\sim 4{\cdot}10^{-7}$, which corresponds to the radius of bending of ~ 100 km. In the present work we describe the first successful attempt to apply the BFND technique for detecting ultra-small residual stress in a thick Si crystal coated with a thin Ni film.

1. Sample Preparation and Mounting



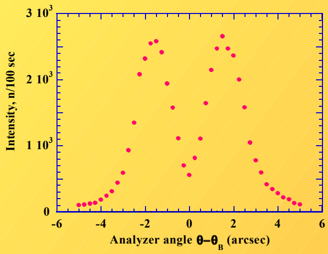
A perfect Si(111) slab-shaped crystal with dimensions 114 mm x 40 mm x 8.19 mm was cut with the 114 mm x 40 mm diffractive surfaces parallel to the (111) crystallographic planes. The diffractive surfaces were polished mechanically, etched, and finally polished chemically. The Front Face (FF), Back Face (BF) and Garland Rocking Curves (FFRC, BFRC, and GRC) were measured before and after coating of one of the diffractive surfaces with a 2000Å thick Ni film using a magnetron sputtering technique. The crystal under study was mounted on the rotation stage without external deformation strain.

2. Experimental Set-up



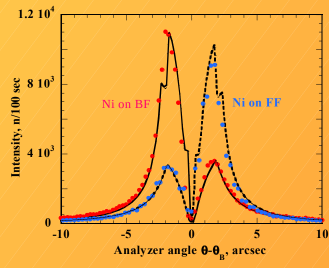
The experiments were carried out using the Double-Crystal Diffractometer at Oak Ridge National Laboratory. The primary beam was diffracted from a Si(111) triple-bounce channel-cut monochromator; the wavelength was $\lambda = 2.59$ Å, and the beam cross-sectional area was 2 cm x 4 cm. The beam was restricted with a fixed vertical 1.8 mm wide Cd slit and the second scanning 4 mm wide slit was mounted directly in front of the detector. In this configuration, the intensity diffracted from different volumes within the crystal was mapped and the transmitted beam was used as a monitor signal for determination of the exact Bragg angle, θ_B, when the rocking curves were measured for positions other than the front face of the crystal.

3. Dynamical Diffraction Measurements on the Substrate



The experimental BFRC (1) obtained from the perfect Si crystal without Ni film (substrate itself) is symmetric with respect to the exact Bragg angle, $\theta - \theta_B = 0$, that according to the dynamical diffraction theory proves the quality of our sample.

4. Dynamical Diffraction Measurements on the Si Crystal Coated with Ni Film



The dramatic changes in the BFRCs have been observed for the same crystal after depositing the 2000 Å Ni film on one of the diffractive surfaces. The BFRCs, measured when the Ni coated surface is set up as the BF and then as the FF, are strongly asymmetrical, and can be considered as the mirror images of each other. The theoretical BFRCs calculated for the dimensionless parameters of deformation, $b \approx 4{\cdot}10^{-4}$ and $b \approx 3.5{\cdot}10^{-4}$, fit quite well to the corresponding experimental rocking curves.

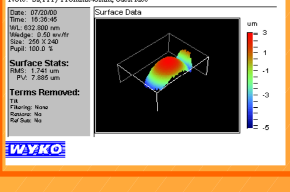
5. Residual Stress in the Ni Film

The parameter $b \approx 4{\cdot}10^{-4}$ corresponds to the relative deformation of the Si crystallographic cells in the vicinity of diffractive surfaces, $|\partial u_z/\partial z| \approx 1.6{\cdot}10^{-6}$, and to the radius of bending, $R_b \approx (H/2b)(\tau/\pi)^2 \approx 19$ km, where τ is the extinction length. The Stoney formula converts the value of R_b to the tension force, f, applied to the film as a result of the substrate deformation:

$$f = ET^2 / [6(1-\nu^2)R_b],$$

where $E \sim 10^{12}$ dyn/cm^2 is the modulus of elasticity, and ν is the Poisson constant of Si.
Thus, the calculated value of f is 90 ± 5 N/m, which can be converted to units of the tensile strength, 427 MPa. This value is below the ultimate tensile strength of annealed bulk Ni, 586 MPa but above the tensile yield strength of that, 345 MPa. The measured tension force, f, is much greater than the sensitivity limit of the BFND technique, which for our set up was ~ 1 N/m; this value can be further optimized.

6. Laser Interferometry



The surface metrology measurements were carried out on the Si diffractive surface using a WYKO laser figure interferometer. The root mean square height variation,1.74, and the maximum peak to valley value, 7.88 µm, do not change after coating the opposite surface with a 2000 Å Ni film. This experiment proves that the conventional laser-based technique cannot be applied in the case of thick substrates.

Summary

The described experiments clearly show that the BFND can be used for residual stress measurements in thin films deposited on the diffractive surface. The BFND works similarly to the SSIOD *in situ* technique, detecting the deformation of the substrate, thus, it is capable to measure residual stress not only in crystalline, likewise the X-ray diffraction technique, but also in any amorphous, polymer, colloidal, mono- and multilayer thin films deposited on the diffractive surface of Si single crystals. The BFND is not an *in situ* technique and it allows evaluating the final product. We, therefore, expect its broad application, particularly in characterization of neutron and X-ray optical devices and even in the semiconductor industry using perfect Si crystals as test samples.